



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Art Unit: 1713 )  
Examiner: Caixia Lu )  
Inventors: Mingwei Xiao, et al. )  
Serial No: 10/553,694 )  
Filed: 08/30/2005 )  
For: A CATALYST FOR ETHYLENE )  
POLYMERIZATION, A PROCESS FOR )  
PREPARING THE SAME AND USE )  
THEREOF )  
Attorney Docket: 8737-000029/NP )

DECLARATION UNDER  
37 C.F.R. §1.132

I, Shijiong Yu, hereby declare as follows:

1. I am one of the inventors named in the above identified patent application.
2. I have a Bachelor degree from the University of Shanghai University of Engineering Science, 1995; (any other degrees).
3. My present position is Senior Engineer at Shanghai Research Institute of Chemical Industry.
4. I understand that prosecution of the present application may be advanced by the presentation of evidence addressing the following:

Catalysis belongs to a field with results being very difficult to be predicted. Catalysts prepared by different processes, even those prepared from the same raw materials by similar processes but in different reaction orders, might exhibit quite different properties, even though they might have the same or similar composition. This is particularly true for complicated polyolefin catalysts, of which composition and structure, including distribution of active sites in the catalyst, might greatly affect their performance.

5. I have participated in the creation of the Experiment Report below.

### Experiment Report

#### Comparative Example 1

##### 1. Treatment of silica carrier:

30 Grams of silica were charged into a fluidized bed, then heated to 600°C and maintained at that temperature for 10 hours under nitrogen flow. Then the silica was cooled to room temperature and was stored for further reaction.

The heat-treated silica was added into 100 ml of hexane solution containing 2.5 ml of triethyl aluminum, and then hexane was evaporated to give a silica carrier having excellent flowability.

##### 2. Preparation of main catalyst component:

To a reactor were charged with 8 ml of 1M solution of dibutyl magnesium in heptane (available from Fluka Co.) and 50 ml of hexane. Then 10 g of the treated silica carrier was added into the reactor. The mixture was stirred for 1.5 hours, and then 5 ml of n-butyl chloride was added thereto. The mixture was heated to 65°C and maintained at that temperature for 0.5 hours. Then to the reactor were added with 0.8 ml of titanium tetrachloride and 1.0 ml of diethyl aluminum chloride. Upon completion of the addition, the mixture was allowed to react for 3 hours, and then solvent was evaporated at 70°C, to give a solid main catalyst component.

##### 3. Polymerization:

The polymerization reaction was carried out in a fluidized bed reactor having a diameter of 100 mm and a height of 1500 mm. First, 100 g of an oven-dried base of particulate polyethylene, 0.8 ml of triethyl aluminum as cocatalyst and 0.1 g of the main catalyst component as prepared in the above step 2 were charged into the fluidized bed reactor. The polymerization was performed at 90°C under a total pressure of 2.0 MPa for 3 hours, with white polyethylene being obtained. The polymerization results are shown in the below Tables 2 and 3.

#### Comparative Example 2

##### 1. Treatment of silica carrier:

The procedure as described in the Comparative Example 1, step 1 was followed.

##### 2. Preparation of main catalyst component:

To a reactor were charged with 5.9 g 20% solution of butyl magnesium chloride in THF/toluene (available from Acros Co.), 50 ml of THF, and 12 g of the treated silica carrier. The mixture was stirred

for 1.5 hours, and then heated to 70°C with stirring to evaporate solvent. During the evaporation, solid component was sampled for analysis and the evaporation was stopped when the content of residual THF in said solid was 8.2 wt.%. A solid component was obtained.

To the resulting solid component were added 70 ml of hexane and 6.4 ml of n-butyl chloride. The mixture was heated to 65°C and maintained at that temperature for 0.5 hours, and then 1.2 ml of titanium tetrachloride and 1.3 ml of diethyl aluminum chloride were added thereto. Upon completion of the addition, the mixture was allowed to react for 3 hours, and then hexane was evaporated at 70°C, to give a solid main catalyst component.

### 3. Polymerization:

The polymerization reaction was carried out according to the procedure as described in the Comparative Example 1, step 3. The polymerization results are shown in the below Tables 2 and 3.

#### Example 9

A solid main catalyst was prepared according to the procedure as described in Example 1, and evaluated in ethylene polymerization under the same conditions as those employed in the Comparative Examples. The polymerization results are shown in the below Tables 2 and 3.

Table 1

	mmolMg/gSiO <sub>2</sub>	Mg/Ti (mol/mol)	Al/Ti (mol/mol)	BuCl/Mg (mol/mol)	Content of Residual THF (wt.%)	Ti (wt.%)
Comparative Example 1	0.80	0.95	1.0	6.0	/	2.49
Comparative Example 2	0.85	1.0	1.0	6.0	8.2	2.76
Example 9	0.89	1.0	1.0	5.8	5	2.50

Table 2 Polymerization Results\*

	Activity (gPE/gCat.)	MI <sub>2.16</sub> g/10min	Bulk Density g/cm <sup>3</sup>
Comparative Example 1	5014	2.1	0.34
Comparative Example 2	6520	4.2	0.35
Example 9	12500	6.5	0.36

\*The polymerization was carried out under conditions as follows: ethylene partial pressure: 0.8 MPa; hydrogen partial pressure: 0.3 MPa; nitrogen partial pressure: 0.9 MPa; polymerization temperature: 90°C; polymerization time: 3 hours.

Table 3 Polymerization Results\*

	Activity (gPE/gCat.)	MI <sub>2.16</sub> g/10min	Bulk Density g/cm <sup>3</sup>
Comparative Example 1	3578	4.1	0.33
Comparative Example 2	4910	9.0	0.32
Example 9	9140	10.9	0.37

\*The polymerization was carried out under conditions as follows: ethylene partial pressure: 0.8 MPa; hydrogen partial pressure: 0.6 MPa; nitrogen partial pressure: 0.6 MPa; polymerization temperature: 90°C; polymerization time: 3 hours.

6. From the data shown above, it can be seen that under the same polymerization conditions, the catalyst according to the invention exhibits an activity about two times higher than that of the catalysts of Comparative Examples 1 and 2. Additionally, the polyethylene obtained by using the catalyst according to the invention has higher bulk density than that of the polyethylenes obtained by using the catalysts of Comparative Examples 1 and 2.

7. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1000 of Title 18 of the United States Code, and that such statements may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

Shijiong Yu  
(Declarant)

Dated: 24, May, 2007